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MRL-TN-528

AR-005-208

AUSTRALIA

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MATERIALS RESEARCH LABORATORY

MELBOURNE, VICTORIA

TECHNICAL NOTE

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GRANULATION OF PYROTECHNIC TRACER COMPOSITION R284T

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ABSTRACT

This technical note describes the development of several methods to granulate the pyrotechnic tracer composition R284T. The ungranulated composition had been found unsuitable for the automatic machine loading and filling of 7.62 mm F7 tracer bullets due to segregation of the ingredients.

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POSTAL ADDRESS: Director, Materials Research Laboratory
P.O. Box 50, Ascot Vale, Victoria 3032, Australia

CONTENTS

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1. INTRODUCTION

Materials Research Laboratory has developed a method to improve the handling characteristics of the pyrotechnic tracer composition R284T made at Explosives Factory Maribyrnong (EFM). The modification was required to enable the automatic loading, at Ammunition Factory, Footscray (AFF), of 7.62 mm tracer bullets for Army. Previous attempts to improve the physical properties of the composition by the addition of a small percentage of flow modifier had proved unsuccessful. MRL investigated a number of alternative methods using granulation techniques to improve the flow properties of this batch. This technical note describes these methods, and the assistance given to EFM to further develop the most appropriate process for the manufacture of this tracer composition.

2. BACKGROUND

Composition R284T (Appendix A) is an American developed filling for 5.56 mm; 7.62 mm NATO; 0.30" and 0.50" calibre tracer ammunition and is currently manufactured in Australia by EFM for use in 7.62 mm F7 tracer rounds. R284T is a ternary mixture consisting nominally of 28% magnesium, 55% strontium nitrate and 17% polyvinylchloride. Because the formulation incorporates no binder or coating on the magnesium fuel, two areas of significant technical risk are associated with this composition.

(i) Severe segregation of the ingredients is evident after mixing, handling or storage of the loose composition (Figs 1(a) and 2(a)). This is probably due to the disparity in the densities, specific surface areas and particle sizes of the ingredients (Appendix B). Automatic hopper feeding and consequent vibration of the composition exacerbates the segregation problem (particularly after the addition of the flow modifier), resulting in variations in charge mass, ingredient proportions and ultimately, poor tracer round performance.

(ii) The use of uncoated magnesium in the composition leads to scoring of the hopper feed shoe on the loading machine and also may, in time, lead to compositional instability problems in the filled tracer rounds [1].

Due to the lack of information regarding US manufacturing techniques, MRL suggested that granulation of the composition would solve most of the more immediate problems related to its use. It should be observed that the techniques described in this technical note were developed on a laboratory scale and should be used only as a guide for the further development of the process.

3. SOLVENT GRANULATION

'Wet' or solvent granulation of pyrotechnic compositions usually involves the addition of a medium to partially dissolve at least one of the ingredients in the mixture. The dampened composition is then forced through a sizing screen and the resulting granules are dried and sieved to remove fines.

Granulation serves two main purposes -

(i) to control the flow properties of the composition and

(ii) to produce discrete and coherent particles of composition, each of which contains the correct proportions of the ingredients.

After consultation with the Occupational Health Advisory Group, Office of Defence Production and reference to the available literature, three solvents were chosen for evaluation in the granulation process - acetone, ethanol and cyclohexanone (Appendix C). It was decided to granulate samples of the composition using each of these solvents to enable evaluation of their relative performance both on the Manurhin loading machine at AFF and at range firings of 7.62 mm tracer rounds at Proof and Experimental Establishment, Graytown.

3.1 Acetone and Ethanol

The strontium nitrate oxidiser used in R284T is soluble in water and only slightly soluble in acetone or ethanol. Water, however, cannot be used to granulate this composition because of the presence of magnesium metal in the formulation [1].

Although it was expected that the granules resulting from the use of acetone or ethanol would be physically weak because of the low solubility of strontium nitrate, the advantages of cost and operator tolerance make these two solvents acceptable for use in a large industrial process.

100 g of composition R284T was placed in a vessel and sufficient acetone was added to form a wet paste. After mixing by hand to partially dissolve the oxidiser, the paste was allowed to stand in a fume cupboard for several minutes to evaporate the excess solvent. When the composition had achieved the consistency of damp sand, it was quickly forced through an 18 mesh British Standard Sieve using a plastic plug. The resulting granules were allowed to air dry for thirty minutes and were then lightly brushed through a 14 mesh sieve to break up agglomerates. After stoving, the granules appeared very weak and the presence of fines was evident. It was decided not to attempt to remove these fines because any additional work on the sample would further break up the granules. Although the granulated composition was forwarded to AFF for loading and evaluation, MRL did not consider the acetone technique to be viable. The volatility of the acetone presented handling problems which would be extremely difficult to overcome when dealing with large batches of composition. The use of acetone also involves a significant risk due to its susceptibility to ignition by electrostatic discharge [2].

The technique was repeated on another sample of R284T using ethanol as the solvent. At the request of AFF, the composition was forced through a 36 mesh sieve to produce smaller granules which would be more suitable in the loading machine. The results achieved by this method were very similar to the acetone technique - weak granules and a significant proportion of fines.

3.2 Cyclohexanone

Polyvinylchloride (PVC) is soluble in a range of solvents, all of which are hazardous and toxic [3,4]. It was decided to evaluate one of the least dangerous of these solvents, cyclohexanone, to granulate R284T. Using the appropriate precautions, this approach would exhibit at least two advantages over the acetone or ethanol processes:

(i) by dissolving the PVC, granules of high physical strength could be produced and

(ii) adjustment of the process (and compositional) parameters could result in the coating of the magnesium fuel with PVC, thereby yielding a more stable composition, probably without unduly affecting tracer round performance.

Sufficient cyclohexanone was added to 100 g of R284T to form a damp powder. After hand mixing, the sample was divided in half and 50 g was granulated through an 18 mesh sieve. The remainder of the sample was forced through a 36 mesh sieve, but the screen quickly became clogged as the solvent evaporated and the composition became 'plastic'. Although enough composition was recovered from each sample to enable AFF to load a number of tracer rounds for evaluation, it was apparent that the wet granulation technique using cyclohexanone would be very difficult to scale up as a viable industrial process. Because of the apparent high strength of the granules after curing, it was decided to develop a 'dry' granulation process. 500 g of R284T was placed in a mechanical mixer and enough cyclohexanone was added to achieve the required consistency. After thorough mixing, the damp composition was shaken through a coarse sieve onto a tray to break up the 'cake'. The composition was allowed to air cure in a fume cupboard for forty-eight hours and was then stoved at $60^{o}C$ for one hour. The batch was divided into two samples for dry granulation on (i) a 36 mesh sieve and (ii) a 60 mesh sieve. The 36 mesh sample was easily brushed through the sieve, exhibited good flow properties, no segregation of ingredients and only a small percentage of fines. The 60 mesh sieve, however, produced a poor granulation with segregation and poor flow properties evident. The 'dry' granulation technique showed the most promise, but EFM expressed concern over the use of cyclohexanone on the basis of toxicity and cost.

4. GRANULATION BY THE ADDITION OF BINDERS

Another method of granulating pyrotechnic compositions is by the addition to the formulation of a suitable binder, usually dissolved in solvent. The solution is thoroughly blended into the composition and the mixture is sized through an appropriate screen and dried in the normal way. Generally, the addition of a binder will modify not only the physical properties of the composition but also its pyrotechnic performance. Some consequences of this approach might be the sticking of the composition on punches and dies in the loading machine and changes to the explosive sensitivity of the composition; its ignition characteristics; burning rate; light output, compatibility and stability. After consideration of these and other factors, two binders were selected for trial granulations: acaroid resin and nitrocellulose.

4.1 Acaroid Resin

Acaroid Resin is an alcohol soluble plant secretion which is used in pyrotechnics technology mainly as a binder and protective coating for magnesium powder. Formulations containing acaroid resin often have enhanced ignition characteristics and faster burning rates. It is normal to coat the magnesium with between 3% and 6% acaroid resin before blending into the composition but, in this application where the composition had already been manufactured, it was decided to add a much smaller percentage to achieve granulation without adversely affecting the overall performance of the composition.

In order to disperse evenly a small percentage of the resin into the tracer composition, whilst achieving the consistency required for wet granulation, it was decided to add the resin from alcohol solution. A sample of R284T was weighed out and ethanol was added and mixed in until the required consistency was achieved. The volume of solvent added was noted. It had been arbitrarily decided to evaluate granulations of the composition using (a) 0.5% and (b) 1.0% acaroid resin content. In order to achieve these concentrations, two solutions of resin in ethanol were made up - one of 3% and the other of 6% acaroid resin. By adding to each sample of composition the same volume of solution as that required to produce the correct consistency, the percentage and dispersion of the resin could be closely controlled. A sample of R284T was prepared and sufficient 3% resin/alcohol solution added to produce a 0.5% resin content when dry. This mixture was placed on a 36 mesh sieve and hand granulated. After stoving, the granules exhibited good flow properties and a small percentage of fines. Another 0.5% resin content sample was prepared and stoved for dry granulation. The 'cake' was broken up and forced through a 36 mesh sieve using a rubber plug. The resulting granules flowed well, but a large percentage of fines was evident.

Both the wet and dry granulation techniques were repeated on samples having 1.0% acaroid resin content. AFF suggested that granules produced on a 25 mesh sieve would probably be best for use in the Manurhin machine, therefore both these samples were made in this way. The 25 mesh granules produced by either the wet or dry technique with 1.0% resin content exhibited good flow characteristics; a small percentage of fines and little apparent tendency to segregate. Because the granules showed lower physical strength than those produced by the cyclohexanone process (though better than either acetone or ethanol), it is suggested that this technique be further developed using higher concentrations of resin.

4.2 Nitrocellulose

Nitrocellulose (NC) is used as a binder in a number of American pyrotechnic compositions. It was decided to evaluate brushing grade NC lacquer, RD 1198B, thinned with acetone, as a granulation binder for the tracer composition. RD 1198B contains 21% NC (together with 10% tritolyl phosphate) dispersed in a mixture of solvents. The dry granulation method appeared more suited for development as a large scale production process, therefore samples of R284T with (a) 0.2% and (b) 0.4% NC were prepared using the technique previously described. Each 'cake' was dried and forced through a 25 mesh sieve. Of the two samples, the 0.2% NC composition produced the best granulation. The 0.4% sample produced a very hard 'cake' upon drying and this required much force to push it through the sieve, resulting in an unacceptable percentage of fines in the granulation.

5. PRELIMINARY SENSITIVITY TESTING

Because the addition of binders is likely to affect the sensitiveness of the composition to ignition by external stimuli, samples of -

(i) the cyclohexanone granulation

(ii) 1% acaroid resin granulation

(iii) 0.2% NC granulation and

(iv) 0.4% NC granulation

were prepared for Rotter Impact and Mallet Friction Testing. The results appear in Table 1. It was considered inappropriate to test the unmodified composition because, due to the segregation of the ingredients, the results would be questionable. It should be noted that, whichever method is selected for large scale production of tracer composition R284T, samples should be subjected to full Safety Certificate and Thermal Stability testing.

6. CONCLUSION

MRL has modified samples of the tracer composition R284T by a number of alternative methods to improve its handling characteristics (Figs 1(b) & 2(b)). The granulated samples have been supplied to AFF, enabling the successful machine loading and range firing of 7.62 mm F7 tracer bullets. EFM has undertaken the further development of the most appropriate process for production of this composition.

7. ACKNOWLEDGEMENTS

The author wishes to express his appreciation for the contributions to this work by:

(i) Mr A. Eden, EFM (formerly of AFF)
(ii) Mr D. Williams, ODP
(iii) Mr A. Bridger, EFM
(iv) Mr E. Wanat and Miss D. Bajada, Hazards Assessment, MRL

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3. Properties of Materials used in Pyrotechnic Compositions (1963). Engineering Design Handbook - Military Pyrotechnic Series Part 3. AMPC 706-187.

4. Sax, Irving N. (1963). Dangerous Properties of Industrial Materials, 2nd edn. (Reinhold Publishing Corporation, New York).

TABLE 1

TYPE OF TEST		R284T/CYCLOHEXANONE		R284T/1% ACAROID	
Rotter Impact (F of I; Standard RDX = 80)		F of I 82 Std. Dev. 9 cm Mean Gas Volume: 2 ml		F of I 73 Std. Dev. 6 cm Mean Gas Volume: 2 ml	
	ANVIL	**MALLET**		**MALLET**	
Mallet Test (expressed as %)		**Boxwood**	**Steel**	**Boxwood**	**Steel**
	Softwood	0	0	0	0
	Hardwood	0	0	0	0
Sensitiveness to Friction	Yorkstone	0	0	0	0
	Mildsteel	0	0	0	0
	Naval Brass	0	0	0	0
	Aluminium	0	0	0	0
	Bronze	0	0	0	0

TYPE OF TEST		R284T/0.2% NC		R284T/0.4% NC	
Rotter Impact (F of I; Standard RDX = 80)		F of I 82 Std. Dev. 9 cm Mean Gas Volume: 2 ml		F of I 73 Std. Dev. 7 cm Mean Gas Volume: 2 ml	
	ANVIL	**MALLET**		**MALLET**	
Mallet Test (expressed as %)		**Boxwood**	**Steel**	**Boxwood**	**Steel**
	Softwood	0	0	0	0
	Hardwood	0	0	0	0
Sensitiveness to Friction	Yorkstone	0	0	0	0
	Mildsteel	0	0	0	0
	Naval Brass	0	0	0	0
	Aluminium	0	0	0	0
	Bronze	0	0	0	0

APPENDIX A

R284 Tracer Mixture

COMPOSITION:

Ingredient	parts by wt
Strontium Nitrate	53.7
Polyvinyl Chloride	18.1
Magnesium 50/100	28.2

DRAWING NUMBER:

DE 130790007

PARAMETRIC:

Auto Ignition Temperature: 488°C

Decomposition Temperature: 577°C

Density:
- Bulk 1.26 g/cm^3
- Loading 2.4-3.0 g/cm^3

Fuel-Oxidizer Ratio: 0.53

Heat of Combustion: 7130 cal/g

STABILITY:

Hygroscopicity 95% RH: 26.2 %
50% RH: 0.016 %

Thermal Stability
- loss in wt. 0 %
- Change in Configuration None

REFERENCE/NOTES:

AMPC 706-185
TM9-1910
Ellern
McIntyre
Cabbaje & Ewing

SENSITIVITY:

Card Gap: No Detonation

Electrical Spark: > 8 Joules

Electrostatic:
- Minimum Concentration 1.62 oz/ft^3
- Minimum Energy 0.0028 Joules

Friction:
- Steel Shoe NO REACTION
- Fibre Shoe NO REACTION

Ignition & Unconfined Burning:

	EXPLODED		BURN TIME
Single Cube	Y	NX	24 s
Multiple Cube	Y	NX	27 s

Impact Sensitivity:

BoE 3.75 in.

OUTPUT:

Burn Time: 4.72 s/cm

Critical Diameter: 0.53 metre
Critical Height 15 cm

High Explosive Equivalency:
PA Method 8 %

USE: 5.56 MM Round; 0.30 Cal Round
7.62 MM NATO Tracer Bullet
0.50 Cal M1 Cartridge

APPLICATION: Main Tracer Charge

STORAGE:	NATO	DoD
Hazards Class (Q/D)	1.1	7
Compatibility	G	A

APPENDIX B

Magnesium Powder type III (atomized), granulation II to MIL-M-382.

Nominal size	:	40 - 80 mesh (US sieve sizes)
Bulk density	:	0.55 - 0.65 g/cc

Theoretical Maximum Density (TMD) : 1.74 g/cc

Strontium Nitrate (non-hygroscopic) grade A or B to MIL-S-20322 (B)

Nominal size	:	(A)	95% minimum passes US number 100 sieve
	:	(B)	99.9% minimum passes US number 50 sieve 70% minimum retained on US number 140 sieve
T.M.D.	:		2.986 g/cc

Polyvinyl Chloride Powder to MIL-P-203071 (MU)

Nominal size	:	95% minimum passes US number 100 sieve 30% maximum passes US number 325 sieve
Bulk density	:	0.40 - 0.60 g/cc

APPENDIX C

Solvent	Formula	Flash Point	Toxicity (Threshold Limit Value)	Fire Hazard
Acetone	CH_3COCH_3	-18^oC	1000	Dangerous, when exposed to heat, flame or electrostatic discharge.
Ethanol	CH_3CH_2OH	16^oC	1000	Dangerous, when exposed to heat or flame.
Cyclohexanone	$CO(CH_2)_4CH_2$	64^oC	25	Moderate, when exposed to heat or flame.

FIGURE 1(a)

FIGURE 1(b)

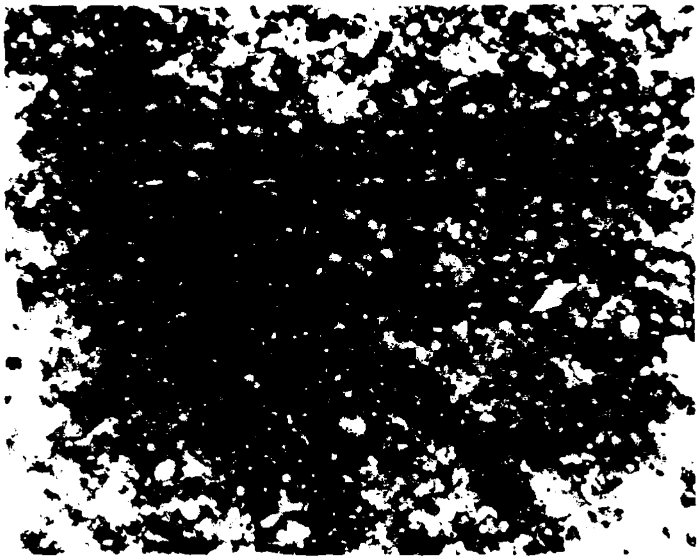

FIGURE 2(a)

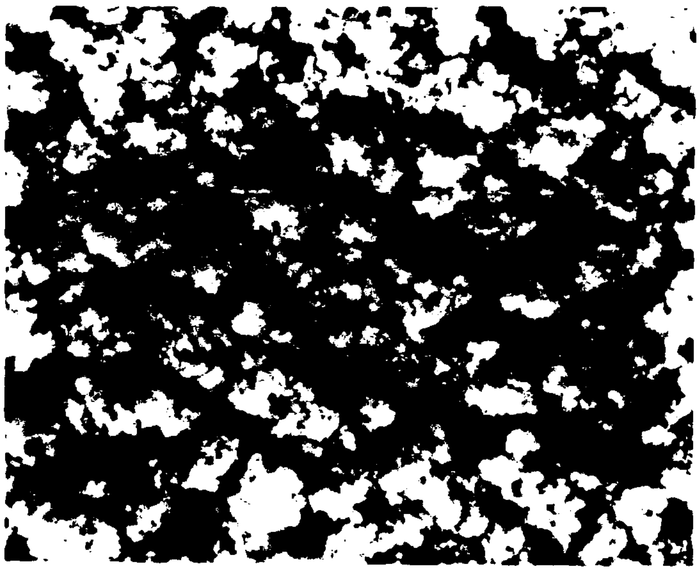

FIGURE 2(b)

SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED

DOCUMENT CONTROL DATA SHEET

REPORT NO.	AR NO.	REPORT SECURITY CLASSIFICATION
MRL-TN-528	AR-005-208	Unclassified

TITLE

Granulation of pyrotechnic tracer composition R284T

AUTHOR(S)

M.A. Wilson

CORPORATE AUTHOR

Materials Research Laboratory
PO Box 50,
Ascot Vale, Victoria 3032

REPORT DATE	TASK NO.	SPONSOR
March 1988	ARM 99/531	DSTO
FILE NO.	**REFERENCES**	**PAGES**
G6/4/8-3500	4	15

CLASSIFICATION/LIMITATION REVIEW DATE

CLASSIFICATION/RELEASE AUTHORITY
Chief of Division,
Explosives, MRL

SECONDARY DISTRIBUTION

Approved for Public Release

ANNOUNCEMENT

Announcement of this report is unlimited

KEYWORDS

Tracers, Tracer composition, Pyrotechnic, Granulation

COSATI GROUPS 0079A

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UNCLASSIFIED

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9 - 88